SYNTHESIS AND CHARACTERIZATION OF SURFACE MODIFIED BANANA TRUNK AS ADSORBENT FOR BENZENE REMOVAL FROM AQUEOUS SOLUTION

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To my beloved family

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ABSTRACT

Petroleum monoaromatics especially benzene contamination in water and wastewater is the major concern in the industry today. Various removal techniques have been studied. The adsorptive removal process is considered as one of the most cost-efficient and feasible methods because it does not require a large amount of energy and additional chemical. In adsorption technology, several problems such as adsorbent cost, adsorption selectivity and adsorbent reusability are the main concerns. Therefore, the potential conversion and modification of agrowaste adsorbent into an effective adsorbent alternative to removing benzene constituent from the wastewater were studied. The banana trunk (BT) was selected as an agrowaste model in the present study. The raw BT (Raw-BT) was first mercerized and this mercerized BT (M-BT) was then reacted with different surfactants, namely cetyltrimethylammonium bromide, CTAB (M-CTAB-BT), 4-(1,1,3,3-tetramethylbutyl)phenyl-polyethylene glycol, Triton X-100 (M-TX100-BT) and sodium dodecyl sulfate, SDS (M-SDS-BT). Surface etherification was also conducted by using 3-chloro-2-hydroxypropyl trimethylammonium chloride, CTA with the mass ratio of 1 and this etherified BT (M-1CTA-BT) was then coated with SDS (M-1CTA-SDS-BT). All the adsorbents were characterized by using a Fourier transform infrared spectrometer, a field emission scanning electron microscope, an x-ray photoelectron spectroscopy analyzer and nitrogen adsorption/desorption analysis. The experimental batch adsorption studies showed that the adsorption capacity increased in the following order: Raw-BT < M-BT < M-CTAB-BT < M-TX100-BT < M-1CTA-SDS-BT. The Langmuir maximum benzene adsorption capacity, $q_{L,max}$ achieved by the M-1CTA-SDS-BT was 468.187×10^{-3} mmol/g. The fundamental adsorption equilibrium and kinetic studies revealed that the benzene adsorption data were fitted well into the Langmuir isotherm and pseudo-second order kinetic models which suggested that the adsorption process was governed by a physical phenomenon. The film diffusion was considered as the rate-limiting step for the overall benzene adsorption process. The adsorbents were chemically stable within the pH range. It was revealed the benzene adsorption was an endothermic and non-spontaneous process. The regeneration study using 50 % ethanol-water as the desorbing agent, demonstrated that the modified adsorbents could withstand five adsorption/desorption cycles without a drastic reduction of adsorption uptake (8.5 - 13.0 %). The BT could potentially be employed as an adsorbent precursor for benzene adsorptive removal from aqueous solution.

ABSTRAK

Pencemaran monoaromatik petroleum terutamanya benzena dalam air dan air sisa adalah kebimbangan utama dalam industri hari ini. Pelbagai teknik penyingkiran telah dikaji. Proses penjerapan merupakan salah satu kaedah yang paling cekap dari segi kos kerana ia tidak memerlukan banyak tenaga dan bahan kimia tambahan. Dalam teknologi penjerapan, beberapa masalah seperti kos bahan penjerap, pemilihan penjerapan dan kebolehan gunapakai bahan penjerap adalah persoalan utama. Oleh itu, potensi penukaran dan pengubahsuaian sisa-sisa pertanian sebagai bahan penjerap pilihan yang berkesan untuk menyingkir juzuk benzena dari air sisa telah dikaji. Batang pisang (BT) telah dipilih sebagai satu model sisa pertanian dalam kajian ini. Batang pisang mentah (Raw-BT) telah dimerserais dahulu dan BT merserais ini (M-BT) telah bertindak balas dengan bahan permukaan yang berbeza seperti setiltrimetilammonium bromida, CTAB (M-CTAB-BT), 4-(1,1,3,3-tetrametilbutil)fenil-polietilena glikol, Triton X-100 (M-TX100-BT) dan natrium dodesil sulfat, SDS (M-SDS-BT). Pengeteran permukaan juga telah dijalankan dengan menggunakan 3-kloro-2-hidroksilpropil trimetilammonium klorida, CTA dengan nisbah jisim 1 dan BT yang melalui proses pengeteran ini (M-1CTA-BT) telah kemudiannya disalut dengan SDS (M-1CTA-SDS-BT) kemudian. Semua bahan penjerap dicirikan dengan menggunakan spektrometer inframerah transformasi Fourier, mikroskop elektron pengimbas pancaran medan, penganalisis spektroskopi fotoelektron sinar-x dan analisis penjerapan/penyahjerapan nitrogen. Eksperimen penjerapan secara kelompok menunjukkan bahawa kapasiti penjerapan meningkat mengikut urutan berikut: Raw-BT < M-BT < M-CTAB-BT < M-TX100-BT < M-1CTA-SDS-BT. Kapasiti penjerapan benzena maksimum untuk model Langmuir yang dicapai oleh M-1CTA-SDS-BT adalah 468.187×10^{-3} mmol/g. Kajian penjerapan telah menggambarkan bahawa seluruh data penjerapan benzena mengikuti model garis sesuhu Langmuir dan model kinetik pseudo kedua yang mencadangkan bahawa proses penjerapan ini mematuhi fenomena fizikal. Peresapan filem dianggap sebagai langkah pembatas kadar bagi keseluruhan proses penjerapan benzena. Bahan penjerap adalah stabil secara kimia dalam julat pH. Kajian ini menunjukkan bahawa proses penjerapan benzena ini adalah proses endoterma yang tidak berlaku secara spontan. Kajian penjana semula bahan penjerap dijalankan dengan menggunakan 50 % larutan etanol-air sebagai agen penyahjerapan. Keputusan kajian ini menunjukkan bahawa bahan penjerap yang diubahsuai dapat bertahan lima kitaran penjerapan/penyahjerapan tanpa pengurangan kapasiti penjerapan yang melampau (hanya 8.5 - 13.0 %). BT adalah sangat berpotensi untuk digunakan sebagai pelopor bahan penjerap untuk menjerap unsur benzena daripada larutan berair.

TABLE OF CONTENTS

CHAPTER	TITLE	PAGE
	DECLARATION	ii
	DEDICATION	iii
	ACKNOWLEDGEMENT	iv
	ABSTRACT	v
	ABSTRAK	vi
	TABLE OF CONTENTS	vii
	LIST OF TABLES	xiii
	LIST OF FIGURES	xv
	LIST OF SYMBOLS	xix
	LIST OF ABBREVIATIONS	xxi
	LIST OF APPENDICES	xxiii
1	**INTRODUCTION**	**1**
	1.1 Research Background	1
	1.2 Problem Statement	3
	1.3 Research Objectives	6
	1.4 Research Scopes	6
	1.5 Thesis Outline	7
	1.6 Summary	7
2	**LITERATURE REVIEW**	**9**
	2.1 Benzene in the Environment	9
	2.1.1 Introduction	9
	2.1.2 Chemical and physical properties of benzene	9

2.1.3 Sources of benzene contamination 11
2.1.4 Health effects caused by benzene 12
2.1.5 Benzene analysis 13
2.2 Benzene Removal Technologies 14
2.2.1 Introduction 14
2.2.2 Bioremediation 14
2.2.3 Wet air oxidation 15
2.2.4 Membrane filtration 15
2.2.5 Adsorption 16
2.3 Benzene Removal by Adsorption 16
2.3.1 Introduction 16
2.3.2 Benzene adsorbents 17
2.3.2.1 Carbonaceous materials 17
2.3.2.2 Silica materials 20
2.3.2.3 Clay materials 23
2.3.2.4 Zeolite and zeolite-like materials 24
2.3.2.5 Polymeric materials 27
2.3.3 Banana trunk as adsorbent precursor 33
2.3.3.1 Introduction 33
2.3.3.2 Origin and properties 33
2.3.3.3 Adsorbent modification 35
2.4 Adsorption Fundamentals 41
2.4.1 Adsorption parameters 41
2.4.2 Adsorption isotherms 43
2.4.3 Thermodynamic parameters 49
2.4.4 Adsorption kinetics 51
2.4.5 Adsorption mechanism 57
2.4.6 Adsorbent regeneration 58
2.5 Summary 59
3 MATERIALS AND METHODS 60
3.1 Introduction 60
3.2 Materials 62

3.3 Adsorbent Synthesis Procedures 63
3.3.1 Introduction 63
3.3.2 Physical modification of agrowaste 63
3.3.3 Surface mercerization of BT adsorbent 64
3.3.4 Chemical modification of BT adsorbents by surfactants 64
3.3.5 Etherification of BT adsorbents 65
3.4 Adsorbent Characterization 65
3.4.1 Microstructure determination 65
3.4.2 Surface chemistry 65
3.4.3 Surface area and pore diameter determination 66
3.5 Benzene Adsorption Procedure 67
3.5.1 Preparation of benzene solution 67
3.5.2 Adsorption experiment 67
3.5.3 Desorption experiment 68
3.6 Analytical Procedures 69
3.6.1 pH determination 69
3.6.2 Benzene concentration determination 69
3.6.3 Surfactant concentration determination 69
3.7 Error analysis 70
3.8 Summary 71

4 RESULTS AND DISCUSSIONS 73

4.1 Introduction 73
4.2 Cationic Surfactant-Coated-Mercerized Banana Trunk Adsorbents 74
4.2.1 Adsorbent characterization 74
4.2.1.1 Surface morphology 74
4.2.1.2 Functional groups 75
4.2.1.3 Chemical state of adsorbent surfaces 76
4.2.1.4 Surface area and pore property 78
4.2.1.5 Benzene adsorption capacity 79

4.2.2 CTAB adsorption characteristics 80

4.2.2.1 Effect of CTAB initial concentrations 80

4.2.2.2 CTAB adsorption isotherm model analysis 81

4.2.2.3 Effect of contact time 83

4.2.2.4 CTAB adsorption kinetic model analysis 84

4.2.3 Batch benzene adsorption studies 86

4.2.3.1 Effects of CTAB loading capacity 86

4.2.3.2 Effect of benzene initial concentration 87

4.2.3.3 Benzene adsorption isotherm model analysis 89

4.2.3.4 Effect of contact time 91

4.2.3.5 Benzene adsorption kinetic model analysis 92

4.2.4 Adsorption mechanism 96

4.2.5 Adsorbent regeneration 98

4.3 Non-Ionic Surfactant-Coated-Mercerized Banana Trunk Adsorbents 99

4.3.1 Adsorbent characterization 99

4.3.1.1 Surface morphology 99

4.3.1.2 Functional groups 101

4.3.1.3 Benzene adsorption capacity 103

4.3.1.4 Surface area and pore property 104

4.3.1.5 Chemical state of the adsorbent surface 104

4.3.2 Batch benzene adsorption studies 107

4.3.2.1 Effect of pH 107

4.3.2.2 Effect of benzene initial concentration and adsorbent dosage 108

4.3.2.3 Isotherm model analysis 109

4.3.2.4 Effect of temperature and contact time 112

4.3.2.5 Thermodynamic parameter analysis 113
4.3.2.6 Kinetic model analysis 114
4.3.3 Adsorption mechanism 119
4.3.4 Adsorption regeneration 120
4.4 Anionic Surfactant-Coated-Etherified Banana Trunk Adsorbents 121
4.4.1 Adsorbent characterization 121
4.4.1.1 Surface morphology 121
4.4.1.2 Functional groups 122
4.4.1.3 Chemical state of adsorbent surfaces 125
4.4.1.4 Surface area and pore property 127
4.4.2 Batch benzene adsorption studies 127
4.4.2.1 Effect of surface etherification 127
4.4.2.2 Effect of pH 129
4.4.2.3 Effect of benzene initial concentration and adsorbent dosage 130
4.4.2.4 Isotherm model analysis 131
4.4.2.5 Effect of temperature and contact time 134
4.4.2.6 Thermodynamic parameters analysis 135
4.4.2.7 Kinetic model analysis 136
4.4.3 Adsorption mechanism 141
4.4.4 Adsorbent regeneration 141
4.5 Comparative Adsorption Studies of Agrowaste Based Adsorbents 143
4.6 Summary 144
5 CONCLUSION AND RECOMMENDATIONS 146
5.1 Summary of Research Findings 146
5.2 Recommendation for Future Research 149
5.3 Concluding Remark 150

REFERENCES **152**

Appendices A - E 176 - 197

LIST OF TABLES

TABLE NO.	TITLE	PAGE
2.1	A summary of benzene properties.	10
2.2	Benzene content in produced water (Neff et al., 2011b).	12
2.3	Maximum allowable BTEX contents in drinking water.	13
2.4	A summary of benzene adsorption capacity of carbonaceous materials.	19
2.5	A summary of benzene adsorption capacity, q (mmol/g) of silica materials.	22
2.6	A summary of benzene adsorption capacity, q (mmol/g) of clay materials.	24
2.7	A summary of benzene adsorption capacity, q (mmol/g) of zeolites and zeolite materials.	26
2.8	A summary of benzene adsorption capacity, q (mmol/g) of polymeric materials.	28
2.9	A summary of conventional adsorbents for VOCs adsorptive removal.	30
2.10	Holocellulose and lignin content of banana pseudo stem, coconut coir, rice straw, rice husk, corncob.	34
2.11	A summary of common adsorbent modification technologies.	36
2.12	Adsorption parameters.	41
2.13	Adsorption isotherm models (Park et al., 2010).	44
2.14	Best fitted adsorption isotherm models.	47
2.15	Thermodynamic parameters of benzene adsorption onto adsorbents.	50
2.16	Reaction based adsorption kinetic models.	55
2.17	Best fitted reaction based adsorption kinetic models and parameters.	56

3.1	Summary of the synthesized banana trunk (BT) adsorbents.	63
4.1	The FTIR wavenumber (cm^{-1}) of various functional groups.	75
4.2	Specific surface area, pore diameter and pore volume of the adsorbents.	79
4.3	Isotherm model parameters for CTAB loading capacity analysis.	82
4.4	Kinetic model parameters for CTAB loading rate analysis.	85
4.5	Isotherm model parameters obtained from benzene adsorption isotherm analysis.	90
4.6	Kinetic model parameters obtained from benzene adsorption kinetic analysis.	93
4.7	FTIR wavenumber (cm^{-1}) of various functional groups.	101
4.8	Specific surface area, pore diameter and pore volume of the adsorbents.	104
4.9	Isotherm model parameters obtained from benzene adsorption isotherm data analysis.	110
4.10	Thermodynamic parameters for benzene adsorptive by M-TX100-BT.	114
4.11	Kinetic parameters obtained from benzene adsorption kinetic data analysis.	117
4.12	FTIR wavenumber (cm^{-1}) of various functional groups.	123
4.13	Specific surface area, pore diameter and pore volume of the adsorbents.	127
4.14	Isotherm model parameters obtained from benzene adsorption isotherm data analysis.	133
4.15	Thermodynamic parameters for adsorption by M-1CTA-SDS-BT.	135
4.16	Kinetic parameters obtained from benzene adsorption kinetic data analysis.	138
4.17	Benzene adsorption capacity comparison among agrowaste adsorbents.	143

LIST OF FIGURES

FIGURE NO.	TITLE	PAGE
3.1	Flowchart of research activities.	61
3.2	Chemical formula and molecular structure of (a) CTAB, (b) Triton-X 100, (c) SDS, (d) CTA, and (e) Pluronic 123.	62
4.1	FESEM image of (a) Raw-BT, (b) M-BT and (c) M-CTAB-BT.	74
4.2	FTIR spectrum of (a) Raw-BT, (b) M-BT and (c) M-CTAB-BT.	76
4.3	An XPS wide scan of (a) Raw-BT, (b) M-BT and (c) M-CTAB-BT.	77
4.4	A high resolution XPS spectrum of the adsorbent: (a) O_{1s}; (b) N_{1s} and (c) C_{1s}.	78
4.5	Benzene adsorption capacity of the synthesized adsorbents (Experimental conditions: initial benzene concentration (C_0), 1.0 mmol/L; contact time (t), 24 h; pH, 7; temperature (T), 303 K; and adsorbent dosage (S/L), 0.5 mg/mL).	80
4.6	Effect of initial CTAB concentrations on CTAB loading capacity (Experimental conditions: contact time (t), 2 h; pH, 7; temperature (T), 303 K; and dosage (S/L), 2 mg/mL).	81
4.7	Isotherm model analysis of CTAB adsorption equilibrium data.	83
4.8	CTAB adsorption rate of the Raw-BT and M-BT at several CTAB concentration (Experimental conditions: initial CTAB concentration (C_0), 0.1, 1.0 and 5.0 mmol/L; pH, 7; temperature (T), 303 K; and dosage (S/L), 2 mg/mL).	84
4.9	Kinetics of CTAB adsorption onto Raw-BT and M-BT adsorbents (Experimental conditions: initial CTAB	

	concentration (C_0), 1.0 mmol/L; pH, 7; temperature (T), 303 K; and dosage (S/L), 2 mg/mL).	86
4.10	Effects of CTAB loading capacity on benzene adsorption capacity (Experimental conditions: initial benzene concentration (C_0), 1.0 mmol/L; contact time (t), 24 h; pH, 7; temperature (T), 303 K; and adsorbent dosage (S/L), 0.5 mg/mL).	87
4.11	Effect of initial benzene concentrations on benzene adsorption capacity (Experimental conditions: contact time (t), 24 h; pH, 7; temperature (T), 303K; and dosage (S/L), 0.5 mg/mL. (b) Isotherm model analysis of benzene adsorption equilibrium data).	88
4.12	Isotherm model analysis of benzene adsorption equilibrium data.	91
4.13	Benzene adsorption rate of Raw-BT, CTAB-BT and M-CTAB-BT and kinetic modeling analysis (Experimental conditions: initial benzene concentration (C_0), 1.0 mmol/L; pH, 7; temperature (T), 303 K; and adsorbent dosage (S/L), 0.5 mg/mL).	92
4.14	Intraparticle/Weber-Morris plots of benzene adsorption kinetic data.	95
4.15	Adsorption performance of M-CTAB-BT as a function of adsorption cycles (Experimental conditions: initial benzene concentration (C_0), 1.0 mmol/L; contact time (t), 24 h; pH, 7; temperature (T), 303 K; and adsorbent dosage (S/L), 0.5 mg/mL).	98
4.16	FESEM image of (a) Raw-BT, (b) M-BT, (c) M-CTAB-BT, (d) M-SDS-BT, (e) M-P123-BT, and (f) M-TX100-BT.	100
4.17	FTIR spectrum of (a) M-CTAB-BT, (b) M-SDS-BT, (c) M-P123-BT, and (d) M-TX100-BT.	102
4.18	Benzene adsorption performance of raw and modified BT adsorbents (Experimental conditions: initial benzene concentration (C_0), 2.5 mmol/L; contact time (t), 24 h; temperature (T), 303 K; and adsorbent dosage (S/L), 1.0 mg/mL).	103
4.19	An XPS wide scan of the M-TX100-BT.	105
4.20	A high resolution XPS spectrum of Raw-BT and M-TX100-BT: (a) O_{1s}; (b) N_{1s}, and (c) C_{1s}.	106
4.21	Effect of pH on benzene adsorption capacity of M-TX100-BT (Experimental conditions: initial benzene concentration (C_o), 2.5 mmol/L; contact time (t), 24 h;	

	temperature (T), 303 K; and adsorbent dosage (S/L), 1.0 mg/mL).	107
4.22	Effect of initial benzene concentrations on benzene adsorption capacity of M-1TX100-BT and M-TX100-BT (Experimental conditions: contact time (t), 24 h; pH, 7; temperature (T), 303 K; and adsorbent dosage (S/L), 0.5 mg/mL and 1.0 mg/mL).	108
4.23	Isotherm model analysis of the equilibrium data: (a) Langmuir and (b) Temkin isotherm models.	111
4.24	Benzene adsorption rate of M-TX100-BT at various reaction temperatures (Experimental conditions: initial benzene concentration (C_0), 2.5 mmol/L; pH, 7; and adsorbent dosage (S/L), 1.0 mg/mL).	113
4.25	Intraparticle/Weber-Morris plot for benzene adsorption by M-TX100-BT at different reaction temperatures.	116
4.26	Kinetic modeling of benzene adsorption onto M-TX100-BT.	118
4.27	Adsorption performance of M-TX100-BT as function of adsorption cycles (Experimental conditions: initial benzene concentration (C_0), 2.5 mmol/L; contact time (t), 24 h; pH, 7; temperature (T), 303 K; and adsorbent dosage, 1.0 mg/mL).	121
4.28	FESEM image of (a) Raw-BT, (b) M-BT, (c) M-1CTA-BT and (d) M-1CTA-SDS-BT.	122
4.29	FTIR spectrum of (a) Raw-BT, (b) M-BT, (c) M-1CTA-SDS-BT and (d) M-1CTA-SDS-BT.	124
4.30	An XPS wide scan of (a) Raw-BT, (b) M-BT, (c) M-1CTA-BT and (d) M-1CTA-SDS-BT.	125
4.31	A high resolution XPS spectrum of M-1CTA-BT and M-1CTA-SDS-BT: (a) O_{1s}; (b) N_{1s} and (c) C_{1s}.	126
4.32	Benzene adsorption capacity, q_e of adsorbents (Experimental conditions: initial benzene concentration (C_0), 2.5 mmol/L; contact time (t), 24 h; temperature (T), 303 K; and adsorbent dosage (S/L), 1.0 mg/mL).	128
4.33	Benzene adsorption capacity of various adsorbents with different mass ratio of adsorbent to CTA (Experimental conditions: initial benzene concentration (C_0), 2.5 mmol/L; contact time (t), 24 h; temperature (T), 303 K; and adsorbent dosage (S/L), 1.0 mg/mL).	129
4.34	Effect of pH on benzene adsorption capacity of M-1CTA-SDS-BT (Experimental conditions: initial benzene concentration (C_0), 2.5 mmol/L; contact time (t), 24 h;	

	temperature (T), 303 K; and adsorbent dosage (S/L), 1.0 mg/mL).	130
4.35	Effect of initial benzene concentrations on benzene adsorption capacity of M-1CTA-SDS-BT (Experimental conditions: contact time (t), 24 h; pH, 7; temperature (T), 303 K; and adsorbent dosage (S/L), 0.5 mg/mL and 1.0 mg/mL).	131
4.36	Isotherm model analysis of equilibrium adsorption data.	132
4.37	Benzene adsorption rate of M-1CTA-SDS-BT at various reaction temperatures (Experimental conditions: initial benzene concentration (C_0), 2.5 mmol/L; pH, 7; and adsorbent dosage (S/L), 1.0 mg/mL).	134
4.38	Intraparticle/Weber-Morris plot for benzene adsorption by M-1CTA-SDS-BT at different reaction temperatures.	139
4.39	Kinetic modeling of benzene adsorption onto M-1CTA-SDS-BT.	140
4.40	Adsorption performance of M-1CTA-SDS-BT as the function of adsorption cycles (Experimental conditions: initial benzene concentration (C_0), 2.5 mmol/L; contact time (t), 24 h; pH, 7; temperature (T), 303 K; and adsorbent dosage (S/L), 1.0 mg/mL).	142

LIST OF SYMBOLS

η_b	-	Water viscosity (0.89 cp)
ρ_s	-	Solid density (g/mL)
$\emptyset$	-	Associate parameter of water (2.60)
ΔG	-	Gibbs free energy change
ΔH	-	Enthalpy change
ΔS	-	Entropy change
a	-	Temkin constant (L/g)
b_T	-	Heat of adsorption (kJ/mol)
C_0	-	Initial adsorbate concentration (mmol/L)
C_e	-	Equilibrium adsorbate concentration (mmol/L)
D_{AB}	-	Molecule diffusivity
D_{eff}	-	Effective diffusion coefficient (m^2/min)
D_{film}	-	Film diffusion coefficient (m^2/min)
D_p	-	Pore diffusion (m^2/min)
D_s	-	Surface diffusion (m^2/min)
ε	-	Polanyi potential
K	-	Thermodynamic constant at equilibrium
k_1	-	PFO equilibrium rate constant (min^{-1})
k_2	-	PSO equilibrium rate constant (min^{-1})
K_F	-	Freundlich constant ($L^n mmol^{n-1}/g$)
k_{id}	-	Intraparticle diffusion constant
k_L	-	External mass transfer coefficient (m/min)
K_L	-	Langmuir constant (L/mmol)
M_B	-	Water molecular weight (18.0 g/mol)
n	-	Adsorption intensity
pH_i	-	Initial adsorbate pH

q	-	Adsorption capacity (mmol/g)
q_e	-	Equilibrium adsorption capacity (mmol/g)
q_{max}	-	Maximum adsorption capacity (mmol/g)
q_t	-	Adsorption capacity at time t (mmol/g)
$r\ \Gamma_e$	-	Retained CTAB loading capacity on the adsorbent (mmol/g)
R	-	Universal gas constant (8.314 J $K^{-1}mol^{-1}$)
R^2	-	Linear coefficient of determination
S/L	-	Adsorbent dosage (solid to liquid ratio, mg/mL)
S_{eff}	-	Slope of the Boyd plot
T	-	Temperature (ºC)
t	-	Time (min)
V	-	Adsorbate volume (L)
V_A	-	Liquid molar volume (m^3/mol)
W	-	Adsorbent Weight (g)
α	-	Initial Adsorption Rate (mmol/g·min)
β	-	Desorption constant (g/mmol)
δ	-	Nernst film thickness
Δq_e	-	Normalized standard deviation for adsorption capacity (%)
$\Delta\Gamma_e$	-	Normalized standard deviation for CTAB loading capacity (%)
ϕ	-	Adsorbent to CTA ratio
χ^2	-	Pearson's chi-squared
Γ_e	-	CTAB loading capacity (mmol/g)

LIST OF ABBREVIATIONS

ACF	-	Activated carbon fiber
BET	-	Brunanuer-Emmet Teller
BT	-	Banana trunk
BTEX	-	Benzene, toluene, ethylbenzene and xylene
CAE	-	Constant analyzer energy
CNT	-	Carbon nanotubes
CPB	-	Cetyl pridinium bromide
CTA	-	(3-chloro-2-hydroxypropyl) trimethylammonium chloride
CTAB	-	Cetyltrimethylammonium bromide
D-R	-	Dubinin-Raduchkevich
FESEM	-	Field emission scanning electron microscope
FID	-	Flame ionization detector
FT-IR	-	Fourier transform infra-red
GC	-	Gas chromatography (GC)
HPLC	-	High performance liquid chromatography
KBr	-	Potassium bromide
NAD	-	Nitrogen adsorption/desorption
NPD	-	Naphthalene, phenanthrene and dibenzothiophene
OPEFB	-	Oil palm empty fruit bunch
PEG	-	Poly ethylene glycol
PFO	-	Pseudo-first order
Pluronic 123	-	Poly(ethylene glycol)-*block*-poly(propylene glycol)-*block* poly(ethylene glycol)
PSO	-	Pseudo-second order
O-SWCNT	-	Oxydized single-walled carbon nanotubes
P-SWCNT	-	Purified single-walled carbon nanotubes
SDS	-	Sodium dodecyl sulfate

SPME	-	Solid phase micro-extraction
TOC	-	Total organic carbon
Triton X-100	-	4-(1,1,3,3-tetramethylbutyl)phenyl-polyethylene glycol
UV	-	Ultra-violet
Vis	-	Visible
XPS	-	X-ray photoelectron spectroscopy

LIST OF APPENDICES

APPENDIX	TITLE	PAGE
A	Ultra-violet visible scan	176
B	Standard calibration curve	178
C	Benzene adsorption capacity screening	181
D	CTAB loading data	182
E	Batch benzene adsorption data	184

CHAPTER 1

INTRODUCTION

1.1 Research Background

In recent decades, many industries such as fossil fuels production, oil and gas refinery, plastic and synthetic rubber manufacturing have been growing extensively every year in order to fulfill the global application and demand (Vidal *et al.*, 2012). As a consequence, water pollution caused by the petroleum production, transportation, processing and application activities has been the major concern all over the world (Tiburtius *et al.*, 2005; Falkova *et al.*, 2016; Li *et al.*, 2016). Petroleum monoaromatic compounds such as benzene, toluene, ethylbenzene and xylene (BTEX) are commonly detected in almost any gasoline and petrol spill in water bodies (Aivalioti *et al.*, 2010). According to a report by the Ohio Department of Health, 18 % of gasoline is made up by BTEX components, by which 11 % of them is benzene component (Ohio Department of Health, 2014). Benzene can enter water bodies by the cracked pipelines, underground fuel tank leakages as well as improper effluent discharge (Sharmasarkar *et al.*, 2000; Seifi *et al.*, 2011a).

Benzene is generally used as either a raw material or a solvent in various chemical and petrochemical related industries (Hindarso *et al.*, 2001; Torabian *et al.*, 2010; Asenjo *et al.*, 2011). It is also one of the volatile organic compounds (VOC) which is colorless and flammable (Bennett and Peters, 1988; Fawell *et al.*, 2003). Many studies have reported that it is highly toxic and carcinogenic to humans (Wibowo *et al.*, 2007; Aivalioti *et al.*, 2012a). It is revealed that a long-term exposure to its compounds at high concentration can cause cancers of the blood forming organs

such as aplastic anemia and leukemia (Weisel, 2010). The stringent regulations have been enforced on its concentration in water bodies due to its toxicity which is harmful to the aquatic environment and human health (Carvalho *et al.*, 2012). According to the World Health Organization (WHO) drinking water guidelines, the maximum permissible its concentration is 0.01 ppm (Gorchev and Ozolins, 2011).

Due to the acute toxicity of the hazardous benzene in water and wastewater, it is utmost crucial to remove it from the waters and wastewaters. Consequently, a wide variety of removal technologies involves chemical, physical and/or biological methods have been investigated. In particular, the most common technologies are chemical or thermal oxidation, bioremediation, volatilization, condensation, membrane separation, and adsorption are developed (Lin and Huang, 1999; Ranck *et al.*, 2005; Farhadian and Duchez, 2008; Fakhru'l-Razi *et al.*, 2009; Aivalioti *et al.*, 2012b; Ali *et al.*, 2012; Carvalho *et al.*, 2012). Among all the conventional removal methods, the adsorption process is suggested to be the most cost-efficient and feasible method because it does not require a large amount of energy and additional chemicals. Besides that, adsorptive removal offers advantages including simple design and operation as well as cheap operating costs (Zytner, 1994; Lin and Huang, 1999; Wibowo *et al.*, 2007; Liang and Chen, 2010; Moura *et al.*, 2011; Yakout and Daifullah, 2013; Ray and Shipley, 2015; Mohammed *et al.*, 2015).

In adsorption technology, activated carbon has been most commonly used as an adsorbent for removing a wide variety of pollutants, such as aromatic organic compounds, heavy metals and dyes, from aqueous solution (Wibowo *et al.*, 2007; Karnib *et al.*, 2014; Sulaymon and Abood, 2014). This is because activated carbon possesses high specific surface area and chemical stability (Hindarso *et al.*, 2001; Su *et al.*, 2010a; Asenjo *et al.*, 2011; Serrano *et al.*, 2011). However, activated carbon is very expensive and not locally available in most of the countries (Daifullah and Girgis, 2003; Seifi *et al.*, 2011c). Besides activated carbon, other materials such as carbon nanotubes (Lu *et al.*, 2008; Su *et al.*, 2010b; Yu *et al.*, 2012), macro-reticular resins (Lin and Huang, 1999), clays (Jaynes and Vance, 1999; Sharmasarkar *et al.*, 2000; Vianna *et al.*, 2005; Nourmoradi *et al.*, 2012), zeolites (Ghiaci *et al.*, 2004; Seifi *et al.*, 2011b), carbon-silica aerogel composites (Dou *et al.*, 2011), diatomite (Aivalioti *et al.*,

2010, 2012a), and membranes (Ohshima *et al.*, 2005; Mukherjee and De, 2016; Uragami *et al.*, 2016) have been employed as adsorbents for the adsorption of benzene and other VOCs.

1.2 Problem Statement

The simple design and operation as well as the capability of adsorbent regeneration, have made the adsorption process as one of the most cost-effective pollutant removal alternatives (Delval *et al.*, 2006; Johari *et al.*, 2013). The active carbon is one of the most effective and conventional adsorbent employed in this process as it can adsorb organic pollutants at a wider spectrum. However, the high affinity toward the adsorbed pollutant molecules causes the difficulty in activated carbon regeneration (Koyuncu *et al.*, 2011). Nowadays, the synthetic adsorbents which are bio-converted from the agricultural wastes (i.e. agrowastes), especially plant fibers, seeds, husks, piths and leaves, have been developed in recently years. This is because agrowastes are cheap, locally available, renewable, natural and biodegradable (Shin and Rowell, 2005; Saman *et al.*, 2014). The applications of raw/unmodified agrowastes such as rice bran, angico saw-dust and peat as agrowaste-based adsorbents for BTEX removal were reported (Adachi *et al.*, 2001; Akhtar *et al.*, 2005; Costa *et al.*, 2012). However, the use of unmodified agrowaste adsorbent has been proven to have low adsorption affinity and selectivity (Ibrahim *et al.*, 2010a; Johari *et al.*, 2016). It is reported that raw agrowastes contain a large number hydroxyl (-OH) groups which has made the agrowastes to be readily functionalized with various chemical functional groups (Kumar *et al.*, 2014). The adsorbent functionalization or modification is conducted to improve their adsorption affinity towards specific pollutants (Tiemann *et al.*, 1999; Delval *et al.*, 2005; Fu and Wang, 2011).

The thermal treatment or carbonization is one of the most common methods used in synthesizing adsorbents (Singh *et al.*, 2003; Demirbas, 2009; Zhou *et al.*, 2015). It was reported that agrowastes such as peach stone, olive stone, date pit, coconut shell, almond shell, and *Moringa oleifera* pods have been employed to produce agrowaste-

based activated carbon for BTEX removal from aqueous solution (Daifullah and Girgis, 2003; Akhtar *et al.*, 2007; Mohammed *et al.*, 2015). A strong binding between the benzene molecules and carbon surface has made it difficult for regeneration and thus increased the material and operating cost (Koyuncu *et al.*, 2011; Zhang *et al.*, 2012b). The alkaline treatment or mercerization process is revealed to create more carboxyl groups and thus produce more potential binding sites on the adsorbent surfaces (Hashim *et al.*, 2012). The mercerization process also resulted in the irregular adsorbent surface as well as increased the adsorbent pore size and surface area by degrading the surface lignin. This leads to better binding effects between the adsorbate and the adsorbent surfaces (Ibrahim *et al.*, 2010a; Koay *et al.*, 2014).

Other chemical modifications such as esterification, acetylation, sulfonation, graft polymerization, impregnation and etherification are also conducted to enhance the adsorption performance towards target adsorbates (Chakraborty *et al.*, 2005; Shin and Rowell, 2005; Alila and Boufi, 2009; Carvalho *et al.*, 2012; Foo and Hameed, 2012b; Song *et al.*, 2013; Teli and Valia, 2013). It was reported that adsorption uptake of BTEX removal relies on the surface hydrophobicity of the adsorbent employed (Seifi *et al.*, 2011b). The surface modification by surfactants (i.e. cationic, anionic and non-ionic) is one of the well-known methods to increase the adsorbents surface hydrophobicity. Agrowastes (e.g. barley and wheat straw, peanut husk, coconut coir pith as well as yeast) functionalized by various cationic surfactants with different molecular weights (e.g. cetylpyridinium bromide (CPB), cetylpyridinium chloride (CPC), cetyltrimethylammonium bromide (CTAB) and hexadecyltrimethylammonium (HDTMA)) prepared using simple impregnation technique are successfully employed in the adsorption of a wide variety of pollutants including oil, dyes and metal ions from waters and wastewaters (Bingol *et al.*, 2004; Namasivayam and Sureshkumar, 2008; Ibrahim *et al.*, 2009, 2010a; Oei *et al.*, 2009; Zhang *et al.*, 2014; Zhao *et al.*, 2014).

The utilization of agrowastes modified with surfactant for removal of benzene or BTEX has so far not been reported. However, inorganic adsorbents such as clay and zeolite modified with cationic surfactants including, benzyldimethyltetradecylammonium (BDTDA), benzyltrimethylammonium (BTMA),

CPB and HDTMA (Koh and Dixon, 2001; Ghiaci *et al.*, 2004; Vianna *et al.*, 2005) have also been employed in volatile organic compounds (VOC) adsorption successfully. It was revealed that the BDTDA modified clay can achieve up to 70 % benzene adsorption uptake of those shown by activated carbon (Koh and Dixon, 2001). Polyethylene glycol (PEG, a type of non-ionic surfactant) was used to modify montmorillonite which showed promising results for the BTEX adsorptive removal process (Nourmoradi *et al.*, 2012). It was suggested that adsorbents modified with non-ionic surfactants may achieve higher BTEX adsorption uptake than that achieved with the adsorbents modified with cationic surfactants. Adsorbents such as bentonite, sawdust, alumina and montmorillonite modified with anionic surfactants (e.g. sodium dodecyl sulfate (SDS) and sodium stearate) were reported for total organic carbon (TOC) and dyes adsorption from aqueous state (Adak *et al.*, 2005; Chen *et al.*, 2010; Ansari *et al.*, 2012; El-dars *et al.*, 2015).

The surfactant-modified agrowastes was investigated as low-cost adsorbents for benzene removal from aqueous solution. The banana trunk (BT) was selected as a model agrowastes in the present study since banana is one of the most cultivated fruits in the tropical region (Hameed *et al.*, 2008). It is generally left unmanaged/decomposing in the plantation estates or disposed at a landfill after fruit harvesting (Sathasivam and Mas Haris, 2010). It is reported that it contains up to 33 % (dry basis) of holocellulose (hemicellulose and cellulose). It also contains a lot of pectin and polyphenols in its extract (Medeiros *et al.*, 2000; Bilba *et al.*, 2007). The high content of holocellulose has made it a potential adsorbent precursor because it is easy to be functionalized to treat the desired pollutant species. It was reported that it was used as adsorbents in adsorptive removal of heavy metal ions such as cobalt (Co(II)), cadmium (Cd(II)), copper (Cu(II)), iron (Fe(II)) and Zinc (Zn (II)), oil and methylene blue (Shibi and Anirudhan, 2005; Hameed *et al.*, 2008; Sathasivam and Mas Haris, 2010; Teli and Valia, 2013). Thus, in the present study, the BT adsorbents were synthesized by surface modification followed by surfactant functionalization to enhance benzene adsorption from aqueous solution.

1.3 Research Objectives

(a) To synthesize low-cost agrowaste based adsorbents by surface mercerization; surfactants modification; and surface etherification.

(b) To characterize synthesized agrowaste based adsorbents.

(c) To investigate the adsorption performance of the synthesized adsorbents towards benzene removal from aqueous solution.

1.4 Research Scopes

The banana trunk (BT) was selected agrowaste based adsorbent precursor. The raw agrowaste adsorbent was firstly modified by surface mercerization. The mercerized adsorbent was coated with various surfactants (i.e. cationic, anionic and non-ionic), namely cetyltrimethylammonium bromide (CTAB), sodium dodecyl sulfate (SDS), Poly(ethylene glycol)-*block*-poly(propylene glycol)-*block*-poly(ethylene glycol) (Pluronic 123) and 4-(1,1,3,3-tetramethylbutyl)phenyl-polyethylene glycol (Triton X-100). The surface etherification of adsorbent was also conducted by introducing the quaternary ammonium groups, namely (3-chloro-2-hydroxypropyl) trimethylammonium chloride solution (CTA) to enhance its surface reactivity towards surfactant coating.

The raw/unmodified and modified adsorbents surface morphologies were characterized by a field emission scanning microscope (FESEM). The existing functional groups on the adsorbent surfaces were determined with the Fourier transform infrared (FTIR) spectrometer and X-ray photoelectron spectrometer (XPS). The Brunauer-Emmet Teller surface area and pore diameters of the adsorbents were also investigated by nitrogen adsorption/desorption analyzer.

The adsorption performance of the surface modified adsorbent towards benzene removal from the aqueous solution was evaluated. The effects of adsorption parameters namely initial pH, initial benzene concentration, adsorbent dosage, temperature and contact time toward benzene adsorption capacity were studied. The isotherm adsorption data were analyzed by using the existing isotherm models namely Langmuir, Freundlich, Temkin and Dubinin-Raduchkevich (D-R) isotherm models, while pseudo-first-order (PFO), pseudo-second-order (PSO), Elovich and Weber and Morris models were applied to investigate the benzene adsorption kinetics. The analysis of the adsorption results led to the proposed adsorption mechanism. The adsorbent regeneration was also investigated.

1.5 Thesis Outline

This research thesis contains seven chapters. Chapter 1 describes briefly of the research background, problem statement, objective and scopes of the study. Chapter 2 presents a critical review on the benzene as pollutants in water, pollutants removal technologies, potential adsorbent precursors, adsorbent modifications as well as technical aspects of adsorption systems. The materials and procedures for adsorbent synthesis and characterization as well as benzene adsorption experiments are discussed in Chapter 3. The research findings are discussed in Chapters 4. The research conclusions and recommendations for future works are presented in Chapter 5.

1.6 Summary

Benzene pollutant existing in the water and wastewater must be removed since it is extremely hazardous and carcinogenic. Flora and fauna as well as humans' health can be seriously affected by its contamination in water. Adsorption is one of the simplest technologies to remove benzene pollutant from aqueous solution using low-cost adsorbents synthesized from agricultural wastes such as the banana trunk. The banana trunk (BT) is cheap and environmentally friendly. In addition, it contains a

large amount of holocellulose which can be easily functionalized. The benzene adsorption by the synthesized adsorbents was evaluated at various experimental conditions followed by the theoretical thermodynamic and kinetic analyses of the experimental adsorption data.

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